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EFFECT OF SUPERPOLISHING ON LASER DAMAGE THRESHOLD

June 1977

Final Report

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AIR FORCE WEAPONS LABORATORY
Air Force Systems Command
Kirtland Air Force Base, NM 87117

This final report was prepared by the Air Force Weapons Laboratory, Kirtland Air Force Base, New Mexico, under Contract F29601-72-C-0033, Job Order ILIR7102. Captain Richard A. House II (DYX) was the Laboratory Project Officer-in-Charge.

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Laser Damage	Bowl-Feed Polishing	Fused Silica
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Ion Polishing	Thresholds	Glasses

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The results of an experimental study to determine the effect of superpolishing techniques on laser induced surface damage are reported. Reduction of surface roughness in five transparent window materials was accomplished using ion polishing and bowl-feed polishing. Surface damage thresholds for these materials were measured at 1.06 μm wavelength and 40.5 nsec pulse width. Comparison of the results was accomplished using the recently determined method of roughness normalization. The ion (OVER)

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20. ABSTRACT

polished samples had greatly improved thresholds, while surface contamination caused a large threshold reduction in the bowl-feed polished samples. Ion polishing was found to be effective in removing surface contamination.

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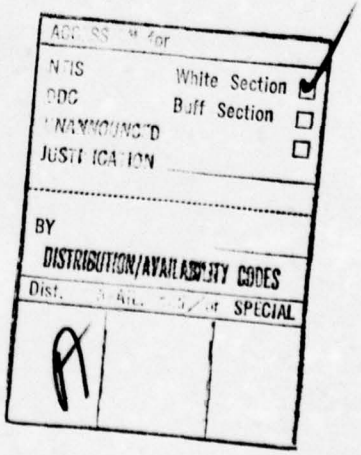

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SECTION I

INTRODUCTION

High-power, Q-switched, solid-state lasers are limited in output power density because of surface damage thresholds exhibited by optical elements. For many transparent materials of interest in the short pulse region (less than 100 nsec), surface thresholds are as much as a factor of 10 lower than corresponding bulk damage thresholds.

It has previously been surmised, based on experimental observations, that laser induced dielectric breakdown is dependent on surface roughness (refs. 1 and 2). Recently, this dependence of threshold on surface roughness (and on other surface parameters) has been systematically investigated (refs. 3 and 4). The purpose of this report is to describe the results of a limited portion of that investigation: namely, the damage testing of transparent dielectric materials that were prepared using the two superpolishing techniques of ion polishing and bowl-feed polishing.

In Section II, the materials and sample preparation procedures are described. Section III describes the laser system, the experimental procedure, and the experimental results. Finally, Section IV contains the conclusions.

SECTION II

SAMPLE PREPARATION

MATERIALS

Five types of commonly used dielectric materials were selected for this study and are listed in table 1. All samples were fabricated as discs of

Table 1

MATERIALS

Material	Index of Refr. (1.06 μm)	Source
BK-7	1.507	Schott "P" quality
Cer-Vit	1.530	Owens-Illinois
ED-2	1.555	Owens-Illinois
ED-4	1.555	Owens-Illinois
Fused Silica	1.449	Corning 7940

diameter 38 mm and of thickness 7.6 mm. The surface flatness figure was $\lambda/10$ (at 632.8 nm). Except for the ED-2 samples, each sample was identically prepared on each of its faces. The ED-2 samples were prepared on only one face, the other face being left in the fine-ground condition.

BK-7 is a boro-silicate crown glass that is commonly used for lenses, windows, and mirror substrates. Cer-Vit is a proprietary vitreous ceramic material that is noted for its ultralow thermal expansion coefficient and is extensively used as mirror substrates. ED-2 and ED-4 are silicate-base glasses of identical composition except that ED-2 is doped with Nd_2O_3 and is the active material for Nd:Glass lasers.

CONTROLLED GRINDING

Preparatory to either ion polishing or bowl-feed polishing, all samples were subjected to a controlled grinding procedure. Controlled grinding is a procedure in which the durations of the various stages are determined by the grinding grit sizes. At each stage of the process, a thickness of material at least three times the previous grit size is removed. The factor of three derives from the empirical rule of thumb that a given grit size produces a disorder zone near the surface (i.e., a zone of dislocations, microcracks, fractures, strain, etc.); and the thickness of this disorder zone is about three times the grit size. Hence, at each stage in the controlled grinding process, an attempt is made to remove the fracture region produced by the previous stage. The final stage in the grinding process was done using a grit size of three micrometers.

ION POLISHING

Following the controlled grinding procedure, all samples to be ion polished were first conventionally polished with jeweler's rouge to remove a 10-micrometer thickness of glass. Each material type was then subjected to ion polishing, the conditions of which were as follows: H^+, Ar^+, and Xe^+ ions; 20 kV and 30 kV ion deposition energies; and $10^{17}/cm^2$ and $10^{18}/cm^2$ total doses. In addition, the ion incidence angle was 70°, and the samples were rotated during bombardment to average the ion planing effect. The incidence angle was chosen for maximum removal of surface atoms (ref. 5). Estimates of the amounts of surface material removed by ion polishing were made (ref. 6), and the results were as follows: for H^+, Ar^+, and Xe^+ ions at a total dose of $10^{17}/cm^2$, the respective estimated thicknesses of material removed were 0.024 μm, 0.24 μm, and 0.060 μm. The thicknesses removed at a total dose of $10^{18}/cm^2$ were a factor of 10 greater.

BOWL-FEED POLISHING

Test samples of all material types were also conventionally polished, following controlled grinding, until they were brought to the "just polished" condition. For each material type, the length of time required to achieve this condition was denoted as "t." Then samples of each material type were bowl-feed polished, using jeweler's rouge, for times of 2t, 3t, 4t, and 5t. For all materials, t was of the order of 20 to 24 hours. Again, except for ED-2, all samples were identically treated on both faces.

Bowl-feed polishing is a process that requires both the lap and the sample to be submerged in a continuously agitated fluid during the entire time of polishing. At the start, the bath is charged with a quantity of polishing compound. Then the process proceeds, without replenishment of compound, until the sample reaches the desired polishing time. Bowl-feed polishing generally produces a significantly smoother surface than that produced by conventional polishing in which new polishing compound is added periodically in a nonsubmerged procedure.

SURFACE ROUGHNESS

The surface roughness, σ, was inferred for each sample by use of a Total Integrated Scattering (TIS) measurement and/or a Talystep stylus measurement. In a TIS measurement, the fraction of an incident light beam scattered by the surface roughness is measured. In a Talystep measurement, a fine-pointed stylus is lightly drawn across the rough surface, the roughness causing measurable displacements of the stylus which is connected to a linear voltage displacement transducer. Both of these techniques are more fully described in references 3 and 4.

DISCUSSION

Ion polishing and bowl-feed polishing are superpolishing techniques. That is, their use makes possible the preparation of surfaces that are smoother than those typically resulting from conventional polishing.

The idea for using ion polishing to smooth optical surfaces and thereby increase laser damage thresholds was originally proposed in 1971 by Dr. Arthur H. Guenther of the Air Force Weapons Laboratory (ref. 7). As a result of this suggestion, Giuliano (ref. 8) damage tested conventionally polished and ion polished sapphire. His preliminary experiment demonstrated that ion polishing did indeed increase thresholds and reduce roughness. Since Giuliano's work was done at low energies (7kV) and high total doses (about $10^{19}/cm^2$), the present work was undertaken to explore the region of higher energies and lower doses.

The comparison of the two superpolishing techniques is of interest for several reasons. In the first place, ion polishing operates on an atomic scale, with surface atoms being removed due to collisional momentum transfer with the incident ions (refs. 9 and 10). Bowl-feed polishing (or any other type of mechanical technique) operates on a macroscopic scale, with surface planing, glass flow, crack-filling, etc., taking place (ref. 11). (In bowl-feed polishing,

though, as contrasted to conventional polishing, the polishing action continually reduces the average particle size so that the surface gets progressively smoother.) Hence, a major difference between ion polished surfaces and bowl-feed polished surfaces is that the former can be intrinsically clean while the latter can be contaminated by impurities. That is, ion polished surfaces could be expected to have damage thresholds more nearly like the pure bulk material than could bowl-feed polished surfaces.

Secondly, ion polishing has a greater potential than bowl-feed polishing for final surface figuring. For instance, in a technique employed at Perkin-Elmer Corporation, the interferogram of an optical surface is used to command a computer controlled ion beam in the final polishing stages. By this means, normally expensive aspheric surfaces have easily been obtained. Such figuring is difficult at best with mechanical polishing techniques.

Finally, ion polishing is a very expensive process compared to mechanical polishing and is not economical for preparing standard flat optical components. Moreover, ion polishing must be preceded by a conventional polishing stage because it cannot be used to polish a fine-ground surface (refs. 9 and 10). Bowl-feed polishing, on the other hand, can be undertaken immediately following the grinding procedure and can be completed on relatively inexpensive equipment.

SECTION III

EXPERIMENTS AND RESULTS

LASER SYSTEM

The laser system used in this study was the oscillator and first amplifier units of a Model VD 640 Nd:Glass pulsed laser system built by the Compagnie General d'Electricite, Marcousis, France. It operated at a wavelength of 1.06 μm and produced TEM_{oo} output pulses of about 400 mJ energy and 40.5 nsec (FWHM) pulse width. The laser beam was focused onto the sample surface, the spot size being 147 μm diameter at the e^{-2} intensity points. Figure 1 depicts the experimental arrangement. The laser was operated at constant input and output, and the energy on target was varied by inserting nonsaturable filters into the beam. Two photodiodes monitored the pulse width and output energy, and a digital readout, pyroelectric detector monitored the energy on target. The primary indicator of a damage event was the observation (by two observers) of a luminous plasma. Auxiliary damage indicators are also shown in figure 1 and explained in detail in references 3 and 4.

PROCEDURE

The damage testing procedure consisted of irradiating a virgin sample site with each laser pulse. A total of 25 shots at each sample was taken, and the data were plotted as in figure 2. In the figure, a damage event is denoted as a "+1" and a nondamage event is denoted as a "-1." The horizontal axis is the measured energy incident on the target. Determination of threshold for any given sample is indicated on the figure; and the energy spread between the highest-energy, nondamage event and the lowest-energy, damage event (i.e., the overlap region) was used to determine the error with which the thresholds were measured. Based on analysis of the experimental data, the threshold energy was ± 19 percent.

ELECTRIC FIELD

As shown in reference 3, the macroscopic optical electric field at the air/target interface is given by (in MKS units)

$$E_{mac} \equiv E_o + E_r = \frac{2}{n+1}\sqrt{\frac{S}{\varepsilon_o c_o}} \simeq \frac{38.8}{n+1}\sqrt{S} \qquad (1)$$

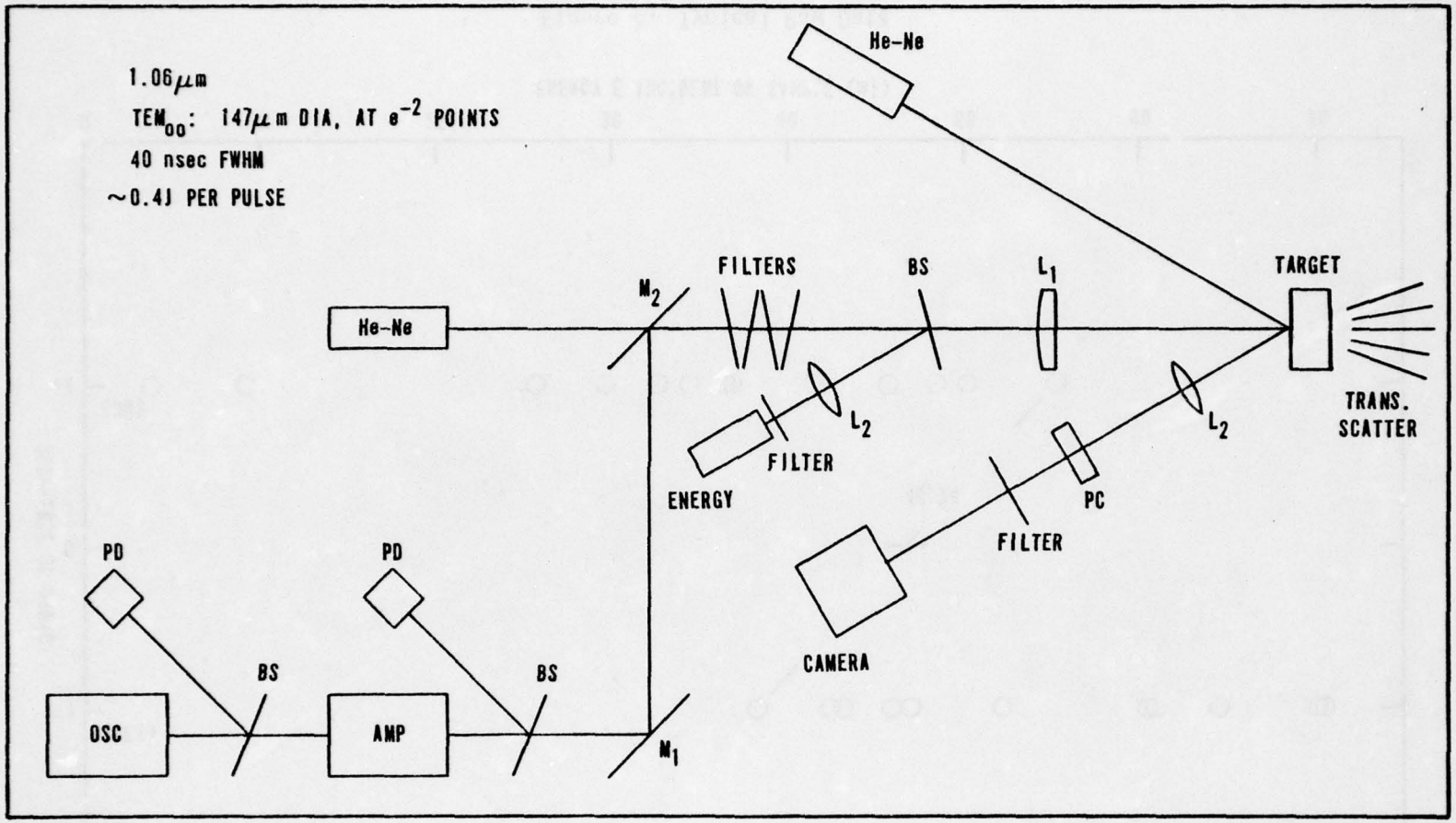

Figure 1. Experimental Arrangement (PD denotes photodiode, BS denotes beam-splitter, M_1 and M_2 are turning mirrors, L_1 is the target lens, L_2 denotes 250 mm lens, PC denotes phase-contrast plate)

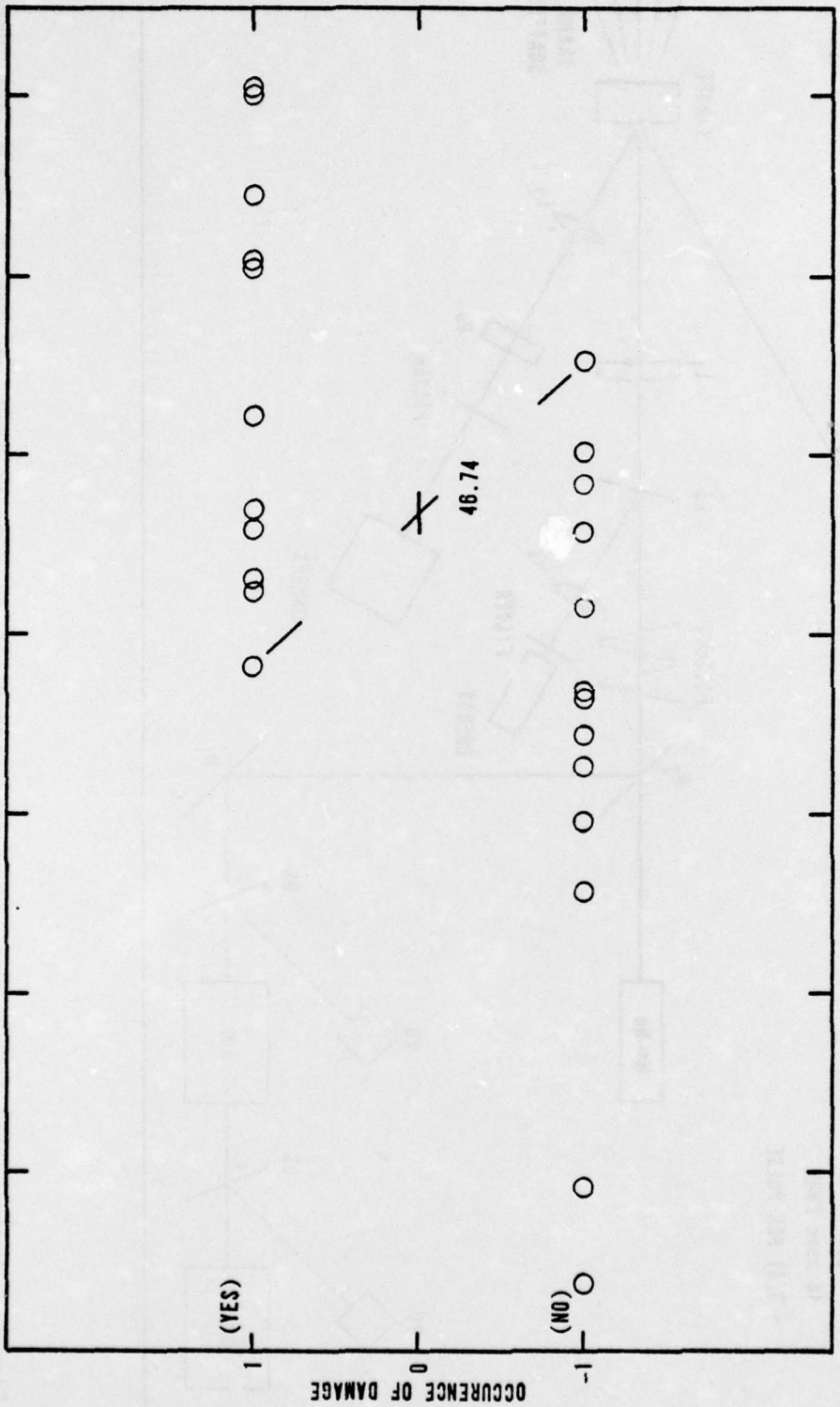

Figure 2. Typical Raw Data

where

E_o = incident optical electric field in V/m
E_r = reflected optical electric field in V/m
n = index of refraction, at 1.06 μm, of the target material
ε_o = 8.8542×10^{-12} F/m (permittivity of free space)
c_o = 2.9979×10^{8} m/s (speed of light in free space)
S = power density (W/m^2) of incident light (Poynting's vector)

The first identity is the definition of the macroscopic field: the vector sum of the incident and reflected fields. This sum, through the Fresnel reflection relationship, results in the factor $\frac{2}{n+1}$. Combining the constants results in the numerical factor 38.8. When S is given in Watts/meter^2 (W/m^2), or in Watts/centimeter^2 (W/cm^2), then the macroscopic field E_{mac} is given in Volts/meter (V/m), or in Volts/centimeter (V/cm). For fused silica, for example, n = 1.449, and $E_r \simeq -0.18\ E_o$. Thus, E_{mac} is approximately $0.82\ E_o$, not E_o. Under conditions of laser induced breakdown, E_{mac} is E_{th}, the threshold total optical electric field that acts upon the target surface and initiates the breakdown event. The precision with which electric field values could be calculated, based on measurements made in this study, was ± 10.4 percent.

A major result of the research reported in reference 3 is the following relationship:

$$E_{th}\sqrt{\sigma} \sim \text{const.} \qquad (2)$$

In this expression, E_{th} is the threshold field and σ is the inferred surface roughness of the target. This expression was shown to be valid for $\sigma \leq 330$ Å. In particular, if one measures the threshold electric fields for a group of samples of various roughnesses prepared by a particular technique, relationship (2) allows one to normalize these threshold fields, via the surface roughness. If one performs this normalization on several groups of samples prepared by several techniques, one can quantitatively compare the fabrication techniques and their effects on the threshold for breakdown.

ION POLISHING RESULTS

A representative selection of ion polished samples was tested for damage susceptibility, and the results are given in table 2.

Table 2

RESULTS FOR ION POLISHING

All Samples Were Previously Polished with Rouge

No.	Mat'l	Ion	Dose (cm^{-2})	E_i (kV)	σ(Å)	E_{th} (MV/cm)	$\sqrt{\sigma}\ E_{th}$
1	BK-7	H^+	10^{17}	20	18	1.43	6.07
2	Cervit	H^+	10^{17}	20	30*	0.55	3.02
3	ED-4	H^+	10^{17}	20	47	1.18	8.09
4	ED-2	H^+	10^{17}	20	15*	0.84	3.25
5	FS	H^+	10^{17}	20	14	1.32	4.94
5	FS	H^+	10^{17}	20	14	1.32	4.94
6	FS	H^+	10^{17}	30	25	1.53	7.65
7	FS	H^+	10^{18}	20	11	2.34	7.76
8	FS	H^+	10^{18}	30	25	1.39	6.95
5	FS	H^+	10^{17}	20	14	1.32	4.94
8	FS	H^+	10^{18}	30	25	1.39	6.95
9	FS	Ar^+	10^{17}	20	31	2.00	11.14
10	FS	Ar^+	10^{18}	30	36	1.68	10.08
11	FS	Xe^+	10^{17}	20	36	1.34	8.04
12	FS	Xe^+	10^{18}	30	31	1.46	8.13
Ref.	FS	--	---	--	13.75	1.60	5.93

*Denotes Talystep measurement

Tabular entries are grouped to facilitate comparison. Samples one through five illustrate the effect due to the type of material, since all these samples were identically polished. Samples five through eight show the effect of varying the dose and ion energy on a single material, fused silica. Samples five and eight through twelve show the effect on fused silica of varying the ion type and of proceeding from low-dose, low-energy to high-dose, high-energy. In the table, E_i is the energy of the ion used to polish the surface and E_{th} is the threshold, macroscopic electric field. The only available roughness measurements for Cer-Vit and ED-2 were Talystep measurements, and these are marked by an asterisk. The reference sample is the smoothest conventionally polished fused silica sample, and it was polished with barnesite. The last column contains the roughness-normalized breakdown fields.

By comparing the electric field thresholds for fused silica with the reference sample—these are the values in the seventh column—it is seen that samples 5, 6, 8, 11, and 12 all had lower thresholds. Only the argon polished samples exhibited higher thresholds than the reference. The conclusion would be that ion polishing is not frequently beneficial. However, a comparison of the normalized fields (the values in the last column) shows that, for fused silica, only sample 5 had a lower threshold than the reference sample. It is not known why sample 5 behaved as it did. The argon polished samples exhibited an increase in normalized field strength of 88 percent, relative to the reference. The xenon polished samples exhibited an increase of 36 percent, and the hydrogen polished samples had an increase of 31 percent.

That ion polishing is very effective in raising the damage threshold can easily be seen by noting that electric field is nearly proportional to the square root of the incident energy or power density. Therefore, a factor of two increase in the electric field threshold is equivalent to a factor of four increase in the more commonly employed threshold units—such as Joules/cm^2 (J/cm^2) or Watts/cm^2 (W/cm^2). For purposes of comparison, table 3 presents corresponding values of electric field, energy density, and power density. The values listed were computed using the index of refraction of fused silica. However, as can be seen from equation (1), the functional dependence on refractive index is weak. Hence, the tabular values are good for all the materials investigated. The first two field values are typical of surface thresholds, while the third value is the experimentally determined value of the bulk threshold in fused silica (ref. 12).

Table 3

CORRESPONDING THRESHOLD UNITS

Electric Field (V/cm)	Energy Density (J/cm^2)	Power Density (W/cm^2)
1×10^6	161	3.98×10^9
2×10^6	645	15.9×10^9
5.2×10^6	4363	107.7×10^9

The reference sample of fused silica was control-ground and conventionally polished with barnesite. Barnesite was selected as the reference polishing compound because it has negligible absorption at 1.06 μm. In 1973, after the ion polished and bowl-feed polished samples had already been fabricated, Boling (ref. 13) reported that the use of jeweler's rouge in the polishing stage led to a sharp reduction in surface damage resistance in optical glasses at 1.06 μm. This reduction in damage resistance was due to contamination of the surfaces by trace amounts of rouge. These trace amounts were small enough to be undetectable by standard chemical analysis techniques, but yet etching restored damage resistance. As can readily be seen by comparison of table 2 and table 4, ion polishing is extremely effective in reducing the level of polishing compound contamination.

As a significant aside, a somewhat related experiment on the ultrasonic cleaning of rouge polished fused silica (ref. 3) showed that long periods of ultrasonic cleaning could also remove most of the rouge contamination. The initial normalized threshold was typical of rouge polished fused silica (table 4) and the final normalized threshold was typical of ion polished material (table 2). Unfortunately, the roughness of the ultrasonically cleaned sample was five times its initial value. Thus, one can conclude that ion polishing is better than ultrasonic cleaning for removing strongly trapped contamination because it simultaneously increases threshold (both normalized and unnormalized) and preserves surface figure and smoothness.

Viewed in relation to the overall experimental program (ref. 3), of which this report covers only a part, ion polishing was very successful. Except for the, as yet, imperfect technique of flame polishing, ion polishing produced fused silica surfaces that had higher thresholds than those produced by conventional polishing, overcoating, etching, and bowl-feed polishing.

BOWL-FEED POLISHING RESULTS

Table 4 gives the results for the bowl-feed polished samples. The first five tabular entries show the material variation, and the fifth through eighth entries show the polishing time variation for fused silica. By comparison of table 4 with table 2, it is seen that the bowl-feed, rouge polished samples were lower in threshold than the ion polished samples (except in the case of Cer-Vit). By comparison of the normalized field values, it is seen that Cer-Vit was also lower in threshold because of the rouge contamination. Not only are the normalized values significantly less than the reference normalized value, but also the fused silica threshold was nearly constant over a wide range in polishing time. The standard deviation of the four values represents a percentage error of 9 percent, which is better than the experimental precision of this study.

A reasonable explanation for the fact that the threshold of fused silica did not depend on polishing time, beyond 48 hours, is that the major portion (probably all of it, in fact) of the in-depth contamination occurs near the start of the polishing process. It is primarily at that time when the surface has a high density of physical traps (cracks, pits, etc.) for the rouge particles.

While it is true that the deleterious effect of rouge on threshold was already known before the present damage tests were made, both the ion polished and bowl-feed polished samples were fabricated before the effect was reported. What the data tables show is that a quantitative measurement of the effect, relative to other polishing techniques, has been achieved (through roughness normalization of threshold).

An additional significant observation from tables 2 and 3 can be made. In the cases of ED-2 and ED-4 (which are glasses of identical composition except for the N_2O_3-doping of ED-2), both polishing processes resulted in higher thresholds for the ED-4. In fact, ED-4 had about twice the electric field threshold of ED-2 (i.e., about four times the energy or power density threshold). This difference may be associated with the increased absorption of 1.06 μm radiation in ED-2 caused by the presence of Nd_2O_3.

Table 4

RESULTS FOR BOWL-FEED POLISHING

Samples Were Polished with Rouge

Material	Time (hr)	σ(Å)	E_{th}(MV/cm)	$\sqrt{\sigma}\ E_{th}$
BK-7	40	30	0.93	5.09
Cer-Vit	46.5	17*	0.55	2.27
ED-4	45	40	0.77	4.87
ED-2	48	21*	0.62	2.84
FS	48	13	0.86	3.10
FS	72	13	0.94	3.39
FS	96	11	0.91	3.02
FS	120	18	0.86	3.65
FS (Ref.)	--	13.75	1.60	5.93

*Denotes Talystep measurement

SECTION IV

CONCLUSIONS

ION POLISHING

In terms of the actual threshold values, the results for ion polishing of fused silica were somewhat disappointing. Three of the hydrogen polished samples and both of the xenon polished samples exhibited a decrease in threshold compared to the reference sample. However, the roughness-normalized breakdown values showed that all three ions used resulted in improved damage thresholds. This result is consistent with the expectation that ion polished surfaces should have a reduced level of mechanical damage and a reduced level of surface contamination. In this work, ion polishing was extremely effective in reducing the surface contamination level. This conclusion is supported by the fact that the samples had been rouge polished prior to being ion polished, and yet the overriding deleterious effect of rouge absorption at 1.06 μm was essentially eliminated. With respect to other commonly used surface preparation techniques such as overcoating with thin dielectric films and etching, ion polishing was clearly superior in raising damage resistance. Surface thresholds as high as 45 percent of bulk threshold were obtained. It was also pointed out, however, that not only is the process expensive in itself, but also it can be used only on a previously polished surface. In addition, for computer controlled surface finishing, ion polishing must be carried out at near normal incidence and probably at lower bombardment energies. Both of these latter conditions imply a large increase in processing time. Lower energies mean fewer removed surface atoms per incident ion, and near normal incidence means that most momentum transfers are directed *into* the target so that surface atoms are ejected from the material with only low probability. Thus, ion polishing may be an inappropriate technique to use on any but specialized, nonstandard optical components.

BOWL-FEED POLISHING

In general, the presence of rouge contamination in the bowl-feed polished samples caused a reduction of threshold. For fused silica, the reduction with respect to the reference sample was about 45 percent. With respect to the ion polished samples, the reduction was about 59 percent. There was no dependence

of threshold on polishing time, within experimental accuracy, in the range of 48 to 120 hours. For all material types tested, the bowl-feed polished samples had lower thresholds than the ion polished samples due to the presence of rouge contamination.

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